

Small Angle Intensities from Double Reflections in Powder Samples*

BY B. E. WARREN

Massachusetts Institute of Technology, Cambridge, Massachusetts, U.S.A.

(Received 17 February 1959 and in revised form 6 April 1959)

A small angle intensity is to be expected from double reflections in any powder sample comprising small grains with random orientation. The inter-grain contribution is due to an hkl reflection in one grain, followed by the same hkl reflection in another grain. The intra-grain contribution arises from a hkl reflection followed by a $\bar{h}\bar{k}\bar{l}$ reflection in the same grain. The inter-grain intensity is independent of grain size and falls off inversely as the scattering angle. The intra-grain intensity depends upon grain size, and falls off rapidly with scattering angle. Equations are obtained for both types of contribution. Cold work in a sample breaks up the original grains into small sub-grains with a small variation in orientation. The enhanced inter-grain contribution from these groups of sub-grains can produce a strong small angle scattering.

1. Introduction

A small angle X-ray scattering from cold worked metals has been observed by Blin & Guinier (1953). It was suggested by Beeman (1957) that the small angle intensities could be due to double Bragg reflections in the powder sample. To be able to discuss quantitatively the effect of cold work on small angle intensities, it is important to first develop a general theory of the small angle intensity due to double Bragg reflections which is to be expected from any powder sample.

We consider that the primary beam is normal to a thin sheet of polygrained sample, in which the grain orientations are random, and the grains small enough so that any beam traverses a large number of grains. Two kinds of double Bragg reflections can contribute to a small angle intensity. Inter-grain scattering consists of an hkl reflection from one grain, followed by the same hkl reflection in a second suitably oriented grain. Intra-grain scattering involves an hkl reflection followed by a $\bar{h}\bar{k}\bar{l}$ reflection in the same grain. We consider first the inter-grain contribution.

2. Inter-grain scattering

The diffraction conditions are illustrated by Fig. 1. The total diffracted power for a reflection hkl from a volume element dV_1 is given by:

$$dP_1 = I_0 K_i dV_1 \quad (1)$$

where

$$K_i = \{e^4 / (m^2 c^4)\} \{\lambda^3 m F^2 / (4v_a^2 \sin \theta_i)\} \quad (2)$$

m is the multiplicity, F the structure factor, v_a the unit-cell volume and θ_i the Bragg angle for the reflection hkl . We omit a Debye temperature factor since the main contribution comes from low order reflec-

* Research sponsored by the U.S. Atomic Energy Commission.

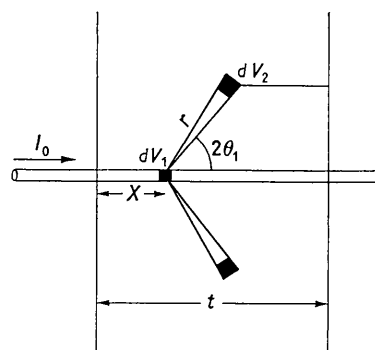


Fig. 1. The volume elements involved in inter-grain scattering in a thin sheet.

tions. The proper polarization factor for a double reflection is put in at the end.

It is convenient to represent the power dP_1 as an intensity distributed uniformly between cones $2\theta_i$ and $2\theta_i + \Delta(2\theta_i)$.

$$dI_r = dP_1 / (2\pi r^2 \sin 2\theta_i \Delta(2\theta_i)).$$

Let dP_2 be the total diffracted power in the reflection hkl from the ring shaped volume $dV_2 = 2\pi r^2 \sin 2\theta_i \Delta(2\theta_i) dr$ irradiated by dI_r .

$$dP_2 = dI_r K_i dV_2 = I_0 K_i^2 dV_1 dr. \quad (3)$$

Fig. 2 represents the concave spherical receiving surface at distance R from the sample. The intersection of the primary beam is at O , and the circle CC is the intersection of the first diffracted cone from volume dV_1 . Each point on circle CC is the center of another circle DD representing the second cone of diffracted radiation. That part of dP_2 which falls in the region between circles B and B' is $dP_2 2d\alpha / 2\pi$, and this is the small angle intensity between cones 2θ and $2\theta + d(2\theta)$ hence

$$dP_2 2d\alpha / 2\pi = dI_{2\theta} 2\pi R^2 \sin 2\theta d(2\theta).$$

Since

$$d(2\theta) = (ds \sin A)/R$$

and

$$ds = 2\pi R \sin 2\theta_i d\alpha / 2\pi$$

we obtain for the small angle intensity

$$dI_{2\theta} = dP_2 / \{2\pi^2 R^2 \sin 2\theta \sin 2\theta_i \sin A\}. \quad (4)$$

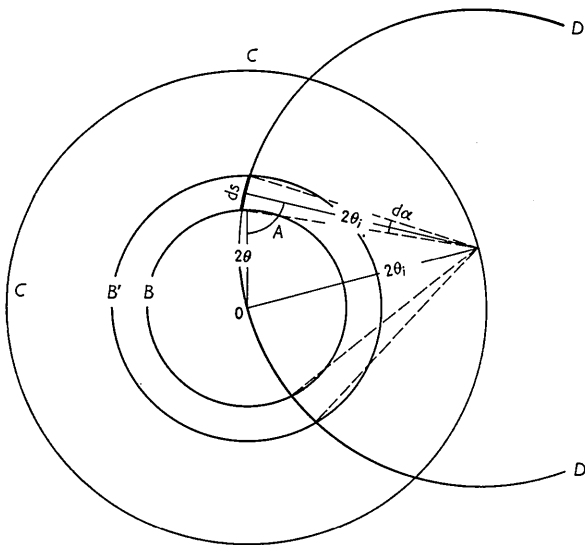


Fig. 2. Intersection of the diffraction cones with the concave receiving surface at distance R from the sample.

From the spherical triangle of sides $2\theta_i$, $2\theta_i$ and 2θ we obtain $\cos A = \tan \theta \cotn 2\theta_i$. Combining this with equations (3) and (4), the small angle intensity becomes

$$dI_{2\theta} = I_0 K_i^2 dV_1 dr / \{4\pi^2 R^2 \sin \theta (\sin^2 2\theta_i - \sin^2 \theta)^{\frac{1}{2}}\}. \quad (5)$$

For $2\theta_i < 90^\circ$, the path length in the sample is $t + 2r \sin^2 \theta_i$, and the absorption factor becomes $\exp[-\mu(t + 2r \sin^2 \theta_i)]$. The integration with respect to r between limits $r=0$ and $r=(t-x)/\cos 2\theta_i$, is readily performed. We now set $dV_1 = a dx$, $P_0 = I_0 a$ and in the square root we drop $\sin^2 \theta$ as negligible compared to $\sin^2 2\theta_i$ since we are only concerned with small angle intensities. The intensity of small angle scattering is given by

$$I_{2\theta} = \frac{P_0 K_i^2 \exp[-\mu t]}{8\pi^2 \mu R^2 \sin \theta \sin^2 \theta_i \sin 2\theta_i} \times \int_0^t \left\{ 1 - \exp\left[-2\mu(t-x) \frac{\sin^2 \theta_i}{\cos 2\theta_i}\right] \right\} dx. \quad (6)$$

The integration with respect to x is readily performed. It is easily shown that the expression obtained applies equally well for $2\theta_i < 90^\circ$ and $2\theta_i > 90^\circ$ if $\cos 2\theta_i$ is replaced by its magnitude $|\cos 2\theta_i|$. Adding the proper polarization factor for a double reflection, the small angle intensity due to a reflection $(hkl)_i$ is given by

$$I_{2\theta} = \frac{P_0 K_i^2 t \exp[-\mu t] (1 + \cos^4 2\theta_i)}{16\pi^2 \mu R^2 \sin \theta \sin^2 \theta_i \sin 2\theta_i} \times \left\{ 1 - \frac{(1 - \exp[-g_i])}{g_i} \right\} \quad (7)$$

where

$$g_i = 2\mu t \sin^2 \theta_i / |\cos 2\theta_i|. \quad (8)$$

It is convenient to express the result in terms of a diffuse intensity in electron units per atom $I(eua)$. In terms of $I(eua)$ and the volume per atom v_0 , we can express the small angle intensity by

$$I_{2\theta} = I_0 \frac{e^4}{m^2 c^4 R^2} \left(\frac{1 + \cos^2 2\theta}{2} \right) I(eua) \frac{at}{v_0} \exp[-\mu t]. \quad (9)$$

Equating (7) and (9), introducing K_i from equation (2) and summing over all hkl reflections, we express $I(eua)$ in terms of the scattering angle ($2\theta^\circ$) in degrees.

$$I(eua) = \frac{1}{(2\theta^\circ)} \left(\frac{e^4}{m^2 c^4} \right) \frac{45\lambda^6 v_0}{32\pi^3 v_a^4 \mu} \times \sum_i \frac{m^2 F^4 (1 + \cos^4 2\theta_i)}{\sin^4 \theta_i \sin 2\theta_i} \left\{ 1 - \frac{(1 - \exp[-g_i])}{g_i} \right\}. \quad (10)$$

The summation over i is performed over all hkl up to $\sin \theta_i = 1$. The small angle intensity due to inter-grain multiple scattering falls off inversely as $(2\theta^\circ)$.

3. Intra-grain scattering

We formulate the problem in terms of a simple restricted case, and later generalize it. We consider a 001 reflection in a cubic crystal containing one atom per cell. Referring to Fig. 3, the horizontal lines are 001 planes and the a_3 vector is vertical. Unit vectors s_0 , s , and s' represent the directions of the primary beam, the first reflected beam, and the final reflected beam. Wave fronts OI and OF perpendicular to the primary and final reflected beams are drawn through the crystal origin O . The first reflection takes place

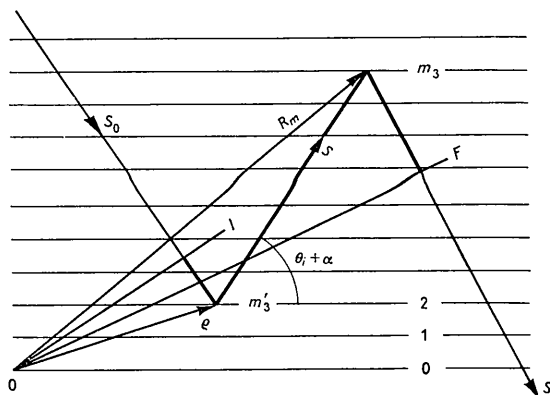


Fig. 3. Double 001 reflection in a single grain. The 001 planes are horizontal and the a_3 axis is vertical. The heavy lines represent the path between an initial wave front OI and a final wave front OF .

from the m'_3 layer, and the second reflection from the atom at position $\mathbf{R}_m = m_1\mathbf{a}_1 + m_2\mathbf{a}_2 + m_3\mathbf{a}_3$. The path travelled between the two wave fronts is

$$\begin{aligned} \text{Path} &= \rho \cdot \mathbf{s}_0 + \mathbf{s} \cdot (\mathbf{R}_m - \rho) - \mathbf{s}' \cdot \mathbf{R}_m \\ &= -(\mathbf{s} - \mathbf{s}_0) \cdot \rho - (\mathbf{s}' - \mathbf{s}) \cdot \mathbf{R}_m. \end{aligned}$$

The primary and first reflected beams each make angles $\theta_i + \alpha$ with the 001 planes, hence $(\mathbf{s} - \mathbf{s}_0) \cdot \rho = (\mathbf{s} - \mathbf{s}_0) \cdot m'_3\mathbf{a}_3$.

If E_0 is the amplitude of the primary beam, E the amplitude of the first reflected beam from the layer m'_3 , and n the number of atoms per unit area in the plane, the usual Fresnel zone treatment gives

$$E = E_0 e^2 f n \lambda / (m c^2 \sin \theta_i). \quad (11)$$

The simplicity of this form results from the fact that we are considering E at a distance r from the plane m'_3 which is small compared to the distance r_0 between the plane m'_3 and the X-ray source, and hence the r dependence of the zone area cancels the $1/r$ dependence of the amplitude per atom. The amplitude of the first reflected beam acting on the atom at \mathbf{R}_m is obtained by summing over all layers m'_3 from $m'_3 = 0$ to $m'_3 = m_3 - 1$. In doing this, we make the approximation that all positions in the layer m_3 receive contributions from all the layers below it. This approximation is bad for reflections at small θ_i , and it could easily result in over-estimating the intra-grain intensity by a factor of 4. If E' is the amplitude in the second reflected beam at a distance R from the crystal, E' is obtained by summing the scattering from each atom at R_m over the whole crystal excluding the zero layer. Since we are considering directions \mathbf{s}' which differ a little from the original directions \mathbf{s}_0 , we can neglect the usual $\pi/2$ and π jumps in phase. The amplitude of the final reflected beam is given by

$$\begin{aligned} E' &= \frac{E e^2 f}{m c^2 R} \sum_{m_1 m_2 m_3} \sum_{m'_3=0}^{m'_3=m_3-1} \\ &\times \exp \left[-\frac{2\pi i}{\lambda} \{(\mathbf{s} - \mathbf{s}_0) \cdot m'_3\mathbf{a}_3 + (\mathbf{s}' - \mathbf{s}) \cdot \mathbf{R}_m\} \right]. \quad (12) \end{aligned}$$

We are interested in a direction \mathbf{s}' which differs by a small fixed amount from \mathbf{s}_0 , so we set $\mathbf{s}' = \mathbf{s}_0 + \Delta$, where Δ is a vector perpendicular to \mathbf{s}_0 and of magnitude $|\Delta| = 2\theta^\circ \pi / 180$. But $(\mathbf{s}' - \mathbf{s}) \cdot \mathbf{a}_1 = \Delta \cdot \mathbf{a}_1$ since $(\mathbf{s} - \mathbf{s}_0) \cdot \mathbf{a}_1 = 0$. Approximating each layer by a disk of radius ρ , the summations over m_1 and m_2 can be expressed as an integral

$$\begin{aligned} \sum_{m_1 m_2} \exp \left[-\frac{2\pi i}{\lambda} \Delta \cdot (m_1\mathbf{a}_1 + m_2\mathbf{a}_2) \right] \\ = \frac{1}{a^2} \int_{r=0}^{\rho} \int_{\varphi=0}^{2\pi} \exp \left[-\frac{2\pi i}{\lambda} |\Delta| r \sin \gamma \cos \varphi \right] r d\varphi dr \\ = (L^2/a^2) (2J_1(x)/x) \quad (13) \end{aligned}$$

where

$$x = \pi^{3/2} 2\theta^\circ L \sin \gamma / (90\lambda).$$

The disk area $\pi\rho^2$ has been equated to L^2 where L is an average grain dimension, and γ is the angle between Δ and \mathbf{a}_3 .

In equation (12) replace E by its value from equation (11). Multiplying by the complex conjugate, and introducing the abbreviation

$$G = I_0 \{e^4 f^2 n \lambda / (m^2 c^4 R \sin \theta_i)\}^2 \{L^4/a^4\} \{2J_1(x)/x\}^2 \quad (14)$$

we obtain I'_c the intensity of the final reflected beam from one crystal.

$$\begin{aligned} I'_c &= G \sum_{m_3=1}^{N_3-1} \sum_{m'_3=0}^{m_3-1} \sum_{n_3=1}^{N_3-1} \sum_{n'_3=0}^{n_3-1} \exp \left[\frac{2\pi i}{\lambda} \right. \\ &\times \left. \{(\mathbf{s} - \mathbf{s}_0) \cdot (m_3 - m'_3 - n_3 + n'_3)\mathbf{a}_3 - \Delta \cdot (m_3 - n_3)\mathbf{a}_3\} \right]. \quad (15) \end{aligned}$$

If M is the number of crystals in the sample, and m is the hkl multiplicity, the number of crystals for which the primary beam makes angles with the (001) planes between $\theta_i + \alpha$ and $\theta_i + \alpha + d\alpha$ is given by $dN = Mm \cos \theta_i d\alpha / 2$. For the powder sample, the intensity $I'(\Delta)$ in a direction differing from the primary beam by an amount Δ , is obtained from

$$I'(\Delta) = \frac{1}{2} M m \cos \theta_i \int I'_c d\alpha. \quad (16)$$

From the Fresnel zone concept, \mathbf{s} and \mathbf{s}_0 make equal angles with the diffracting planes, hence the diffraction vector $(\mathbf{s} - \mathbf{s}_0)/\lambda$ is parallel to \mathbf{b}_3 and in the vicinity of a $00l$ reflection can be expressed by

$$\frac{|\mathbf{s} - \mathbf{s}_0|}{\lambda} = (l + h'_3) |\mathbf{b}_3| = 2 \sin(\theta_i + \alpha) / \lambda \quad (17)$$

$$|\mathbf{b}_3| dh'_3 = 2 \cos \theta_i d\alpha / \lambda.$$

The integration with respect to $d\alpha$ is replaced by an integration with respect to dh'_3 , and to include everything belonging to the reflection $00l$ we integrate from $h'_3 = -\frac{1}{2}$ to $h'_3 = +\frac{1}{2}$.

$$\begin{aligned} \int \exp \left[\frac{2\pi i}{\lambda} (\mathbf{s} - \mathbf{s}_0) \cdot (m_3 - m'_3 - n_3 + n'_3)\mathbf{a}_3 \right] d\alpha \\ = \frac{\lambda |\mathbf{b}_3|}{2 \cos \theta_i} \int_{-\frac{1}{2}}^{+\frac{1}{2}} \exp [2\pi i (m_3 - m'_3 - n_3 + n'_3) h'_3] dh'_3. \quad (18) \end{aligned}$$

Combining equations (15), (16) and (18) we can write

$$\begin{aligned} I'(\Delta) &= \frac{M m G \lambda |\mathbf{b}_3|}{4} \sum_{m_3=1}^{N_3-1} \sum_{m'_3=0}^{m_3-1} \sum_{n_3=1}^{N_3-1} \sum_{n'_3=0}^{n_3-1} \\ &\frac{\sin \pi [(m_3 - m'_3) - (n_3 - n'_3)]}{\pi [(m_3 - m'_3) - (n_3 - n'_3)]} \exp \left[-\frac{2\pi i}{\lambda} \Delta \cdot (m_3 - n_3)\mathbf{a}_3 \right]. \quad (19) \end{aligned}$$

Introduce the new summation indices $j = m_3 - m'_3$ and $k = n_3 - n'_3$. The summations m_3 from 1 to $N_3 - 1$ and m'_3 from 0 to $m_3 - 1$ cover a triangular area in the m_3, m'_3 plane. All points on a 45° line have a constant value for $m_3 - m'_3$. The area can equally well be

covered by a summation over 45° lines and points on each line. Hence we can make the replacement

$$\sum_{m_3=1}^{N_3-1} \sum_{m'_3=0}^{m_3-1} \rightarrow \sum_{j=1}^{N_3-1} \sum_{m_3=j}^{N_3-1}$$

and in terms of the new indices, equation (19) becomes

$$I'(\Delta) = \frac{MmG\lambda|\mathbf{b}_3|}{4} \sum_{j=1}^{N_3-1} \sum_{m_3=j}^{N_3-1} \sum_{k=1}^{N_3-1} \sum_{n_3=k}^{N_3-1} \times \frac{\sin \pi(j-k)}{\pi(j-k)} \exp \left[-\frac{2\pi i}{\lambda} \Delta \cdot (m_3 - n_3) \mathbf{a}_3 \right]. \quad (20)$$

Since all terms are zero except for $j=k$, this reduces to the triple sum

$$I'(\Delta) = \frac{MmG\lambda|\mathbf{b}_3|}{4} \sum_{j=1}^{N_3-1} \sum_{m_3=j}^{N_3-1} \times \exp \left[-\frac{2\pi i}{\lambda} \Delta \cdot \mathbf{a}_3 m_3 \right] \sum_{n_3=j}^{N_3-1} \exp \left[\frac{2\pi i}{\lambda} \Delta \cdot \mathbf{a}_3 n \right].$$

The sums over m_3 and n_3 are readily evaluated and we obtain

$$I'(\Delta) = \frac{MmG\lambda|\mathbf{b}_3|}{4} \sum_{j=1}^{N_3-1} \left[\frac{1 - \cos(2\pi/\lambda)\Delta \cdot \mathbf{a}_3(N_3-j)}{1 - \cos(2\pi/\lambda)\Delta \cdot \mathbf{a}_3} \right]. \quad (21)$$

With the abbreviation $\varepsilon = 2\pi\Delta \cdot \mathbf{a}_3/\lambda$, the summation in equation (21) is readily evaluated.

$$\sum_{j=1}^{N_3-1} \frac{1 - \cos(N_3-j)\varepsilon}{1 - \cos \varepsilon} = \left[N_3 - \frac{1}{2} - \frac{\sin(N_3 - \frac{1}{2})\varepsilon}{\sin \varepsilon} \right] \frac{1}{2 \sin^2 \varepsilon/2}.$$

Replacing $(N_3 - \frac{1}{2})$ by N_3 , making the approximation that ε is small, and adding a polarization factor and an absorption factor, we obtain

$$I'(\Delta) = \frac{MmG\lambda|\mathbf{b}_3|}{12} \left(\frac{1 + \cos^4 2\theta_i}{2} \right) \exp[-\mu t] N_3^3 \Phi(z) \quad (22)$$

where

$$\Phi(z) = \frac{6}{z^2} \left(1 - \frac{\sin z}{z} \right)$$

and

$$z = \pi^2 2\theta^\circ L \cos \gamma / (90\lambda).$$

The function $\Phi(z)$ is defined so that $\Phi(0) = 1.0$, and γ is the angle between Δ and \mathbf{a}_3 .

The small angle scattering can also be expressed in terms of a diffuse intensity $I(eua)$ in electron units per atom.

$$I'(\Delta) = I_0 \frac{e^4}{m^2 c^4 R^2} \left(\frac{1 + \cos^2 2\theta}{2} \right) \times I(eua) M N^3 \frac{v_a}{v_0} \exp[-\mu t]. \quad (23)$$

The number of crystals is M , the number of unit cells per crystal is N^3 , and (v_a/v_0) the number of atoms per cell is included to cover the case where the cell contains more than one atom. Equating (22) and (23) and letting $N_1 = N_2 = N_3 = N$, $|\mathbf{b}_3| = 1/a$, $n = 1/a^2$ and $v_a = a^3$, we obtain the small angle intensity in electron units per atom

$$I(eua) = \left(\frac{e^4}{m^2 c^4} \right) \frac{\lambda^3 v_0 L^4}{24 v_a^4} \left[\frac{m f^4 (1 + \cos^4 2\theta_i)}{\sin^2 \theta_i} \right] \times \left[\frac{2J_1(x)}{x} \right]^2 \Phi(z). \quad (24)$$

To generalize for a crystal containing more than one atom per cell, we replace f by the cell structure factor F , and recognize that v_0 the volume per atom and v_a the cell volume are no longer equal. Equation (24) gives the contribution from one reflection, and we must now sum over all possible hkl reflections. The final equations for the intra-grain contribution are:

$$I(eua) = \left(\frac{e^4}{m^2 c^4} \right) \frac{\lambda^3 v_0 L^4}{24 v_a^4} \sum_i \frac{m_i F_i^4 (1 + \cos^4 2\theta_i)}{\sin^2 \theta_i} \times \left[\frac{2J_1(x)}{x} \right]^2 \Phi(z) \quad (25)$$

where

$$x = \pi^{3/2} 2\theta^\circ L \sin \gamma / (90\lambda)$$

$$z = \pi^2 2\theta^\circ L \cos \gamma / (90\lambda).$$

If φ is an angle of azimuth in the receiving plane normal to the primary beam

$$\cos \gamma = \cos \theta_i \cos \varphi \quad (26)$$

and hence x and z are functions of the reflection i and the azimuth angle φ . For a powder pattern it is necessary to average $I(eua)$ from equation (25) over all values of φ and hence a rigorous evaluation would be very tedious. However one or two reflections usually contribute more than all the others combined, for example, the (111) reflection in a FCC metal. Hence for the FCC case we use θ_{111} in equation (26) and take $[2J_1(x)/x]^2 \Phi(z)$ outside of the summation. In terms of the variable $(2\theta^\circ L/\lambda)$, an average value of $[2J_1(x)/x]^2 \Phi(z)$ is obtained by averaging over 10° intervals in φ throughout one quadrant.

It is possible to reduce equation (25) to a much simpler approximate expression by representing $[2J_1(x)/x]^2$ and $\Phi(z)$ as Gaussian functions matched at the half maximum height. If in addition we use an average value in the exponent,

$$\langle \cos^2 \gamma \rangle = \langle \cos^2 \varphi \rangle \cos^2 \theta_i = 0.5 \cos^2 \theta_i,$$

we obtain

$$I(eua) = \left(\frac{e^4}{m^2 c^4} \right) \frac{\lambda^3 v_0 L^4}{24 v_a^4} \sum_i \left[\frac{m_i F_i^4 (1 + \cos^4 2\theta_i)}{\sin^2 \theta_i} \right] \times \exp \left[-(1 - 0.20 \cos^2 \theta_i) \left(\frac{L 2\theta^\circ}{31.3\lambda} \right)^2 \right]. \quad (27)$$

Although equation (27) is much simpler for purposes of computations, the tails of the curves are cut off more sharply than they should be, as is usually the case when Gaussian approximations are used.

4. Discussion

To illustrate the magnitude of the small angle intensity to be expected from multiple scattering in any powder sample, we take the specific case of a copper sample using Co $K\alpha$ radiation. The sums of equations (10) and (25) include the reflections 111, 200, 220, 311, 222 and 400. The results are presented in Fig. 4. The inter-grain contribution from equation (10) is shown by the dashed lines for sample thicknesses $t=1/\mu$, $t=2/\mu$, and $t=3/\mu$. The intra-grain contribution from equation (25) is shown for a series of grain sizes from $L=50 \text{ \AA}$ to $L=800 \text{ \AA}$. For a scattering

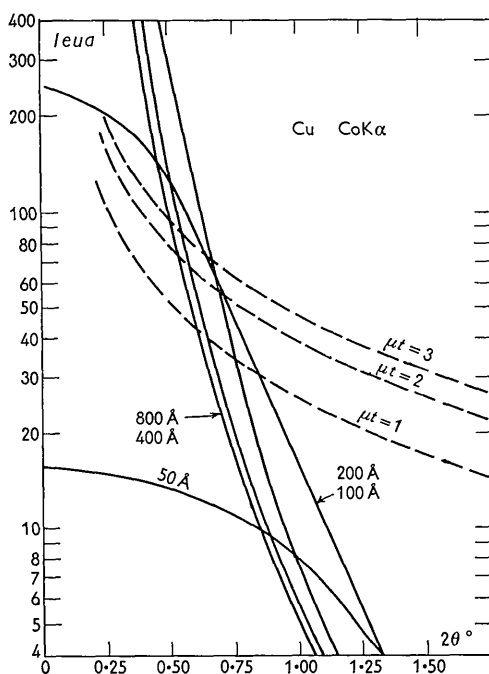


Fig. 4. The intensity in electron units per atom of small angle scattering due to double Bragg reflections in a sample of polygrained copper using Co $K\alpha$ radiation. The dashed lines represent inter-grain scattering and the full lines represent intra-grain scattering for several grain sizes.

angle $2\theta=1$ degree, and a sample thickness $t=2/\mu$, the inter-grain intensity is 40 electron units per atom. Although small, this intensity is large enough to measure with standard small angle scattering apparatus. It is of the same order of magnitude as the absolute values obtained from cold worked and annealed nickel by Blin & Guinier (1953).

When a metal is cold worked, there is a considerable increase in the small angle intensity in the angular range of one or two degrees. This can be partly due to a reduction in grain size and introduction of strains extending the intra-grain contribution out to larger angles. It can also be due to cold work breaking up the larger grains into small sub-grains with slightly varying orientations. This can produce a large increase in the inter-grain contribution, since the first reflected beam now has a much higher probability of meeting suitably oriented grains for the second reflection than was the case for a sample in which neighboring grains have a completely random orientation. Presumably it is the second effect which is of importance in the small angle intensities produced by cold work. The theory of this enhanced inter-grain contribution resulting from cold work has been treated by Nickel, Ogier & Wild (1959).

The intensity distribution in the small angle scattering depends strongly on the correlation in orientation of the fragments or sub-grains produced by cold work. Hence small angle scattering measurements offer the possibility for obtaining information about the correlation in orientation of the fragments or sub-grains, a kind of information not obtainable from line broadening measurements.

It is a pleasure to acknowledge stimulating discussions with Drs A. Franks and V. Gerold, and to thank Dr R. L. Wild for making available in advance a paper on this subject.

References

- BEEMAN, W. W., NEYNABER, R., BRAMMER, W., WEBB, M. & SHARP, G. (1957). *Bull. Fourth Int. Cong. of I. U. Cr.* p. 97.
 BLIN, J. & GUINIER, A. (1953). *C. R. Acad. Sci., Paris*, **236**, 2150.
 OGIER, W. T., WILD, R. L. & NICKEL, J. C. (1959). *J. Appl. Phys.* **30**, 408.